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MENTATION PAGE

Form Approved  
OMB No. 0704-0188

1. SECURITY CLASSIFICATION		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION		3. DISTRIBUTION / AVAILABILITY OF REPORT	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE		Approved for Public Release Distribution Unlimited	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)		5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION		7a. NAME OF MONITORING ORGANIZATION	
Texas A&M University		Texas A&M Research Foundation	
6b. OFFICE SYMBOL (If applicable)		7b. ADDRESS (City, State, and ZIP Code)	
		Box 3578 College Station, Texas 77843	
6c. ADDRESS (City, State, and ZIP Code)		7c. ADDRESS (City, State, and ZIP Code)	
Department of Chemistry College Station, Texas 77843			
8a. NAME OF FUNDING / SPONSORING ORGANIZATION		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER	
Office of Naval Research		N00014-89-J-1214	
8b. OFFICE SYMBOL (If applicable)		10. SOURCE OF FUNDING NUMBERS	
ONR			
8c. ADDRESS (City, State, and ZIP Code)		PROGRAM ELEMENT NO.	
800 North Quincy Street, Code 1513:CF Arlington, VA 22217-5000		PROJECT NO.	
		TASK NO.	
		WORK UNIT ACCESSION NO.	
11. TITLE (Include Security Classification)			
Macrocycles Containing Tin. (Unclassified)			
12. PERSONAL AUTHOR(S)			
Martin Newcomb			
13a. TYPE OF REPORT		13b. TIME COVERED	
Final		FROM TO	
		14. DATE OF REPORT (Year, Month, Day)	
		1991, December 31	
		15. PAGE COUNT	
		6	
16. SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
19. ABSTRACT (Continue on reverse if necessary and identify by block number)			
A summary of project results is reported.			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT			
<input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS			
21. ABSTRACT SECURITY CLASSIFICATION			
Unclassified			
22a. NAME OF RESPONSIBLE INDIVIDUAL		22b. TELEPHONE (Include Area Code)	
Harold E. Gaud		(202)-696-1100	
		22c. OFFICE SYMBOL	
		ONR	

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OFFICE OF NAVAL RESEARCH

Grant N00014-89-J-1214

R & T Code 4135009

Final Report

by


Martin Newcomb  
Department of Chemistry  
Wayne State University  
Detroit, MI 48202

December 31, 1991

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**92-04938**  


## Macrocycles Containing Tin

The project involved the development of methods for the synthesis, purification and characterization of pre-organized macromolecular hosts containing several Lewis acidic tin atoms, and investigations of the potential for such compounds to bind anions and basic donors selectively. The rationale for the study was that macrocyclic species containing Lewis acidic sites should bind anions and donors within the cavity of the macrocycle in a manner analogous to cation binding by "crown ethers" and "cryptands", and that coordinatively saturated tin atoms, which can become pentacoordinate, are good candidates for the Lewis acids. Selective binding properties that correlate to the fit of an anion or donor in the organized binding region of the macromolecular host were expected, and such properties might be exploited for anion separation, anion identification, and catalysis.

A series of macrobicyclic hosts containing two Lewis acidic tin atoms as binding sites was developed, and anion binding was studied by  $^{119}\text{Sn}$  NMR spectroscopy (TR-1, Pub-2). Tricyclic, ditopic hosts containing four Lewis acidic tin atoms were developed (TR-2, Pub-3). X-ray crystal structure determinations of a number of the hosts were obtained (TR-4, TR-5, TR-6, TR-7, TR-8, TR-9, TR-10, TR-11, Pub-5). Solid complexes of macrobicyclic hosts binding anions were isolated and studied by X-ray crystallography and solid state  $^{119}\text{Sn}$  NMR spectroscopy (Pub-1). Force field parameters for compounds containing tetrahedral tin atoms were developed and evaluated (TR-3, TR-12, Pub-4). The structures of the macrobicyclic hosts were studied by molecular mechanics calculations, and the results were compared to the crystal structures (TR-13, Pub-5).



Accession For	
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Justification	
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Distribution	
Availability	
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A-1	

## List of Technical Reports

1. "Macrocycles Containing Tin. The Preparation of Macrobicyclic Lewis Acidic Hosts Containing Two Tin Atoms and  $^{119}\text{Sn}$  NMR Studies of Their Chloride and Bromide Binding Properties in Solution", Michael T. Blanda, John H. Horner, Martin Newcomb, May 24, 1989
2. "Macrocycles Containing Tin. Ditopic, Tricyclic. Lewis Acidic Hosts with Four Binding Sites", Michael T. Blanda and Martin Newcomb, August 30, 1989
3. "A Preliminary Set of Parameters for Force Field Calculations of Compounds Containing Tetrahedral Tin", John H. Horner and Martin Newcomb\*, May 24, 1990
4. "X-Ray Crystal Structure of 1,1,6,6,11,11,16,16-Octaphenyl-1,6,11,16-tetrastannaeicosane", Martin Newcomb and John H. Horner, November 7, 1990
5. "X-Ray Crystal Structure of 1,7-Diphenyl-1,7-distannabicyclo[5.5.5]heptadecane", Martin Newcomb and John H. Horner, November 16, 1990
6. "X-Ray Crystal Structure of 1,10-Diphenyl-1,10-distannabicyclo[8.8.8]hexacosane", Martin Newcomb, John H. Horner and Joseph P. Riebenspies, November 21, 1990
7. "X-Ray Crystal Structure of 1,12-Diphenyl-1,12-distannabicyclo[10.10.10]dotriacontane", Martin Newcomb and John H. Horner, November 26, 1990
8. "X-Ray Crystal Structure of 1,7-Dichloro-1,7-distannabicyclo[5.5.5]heptadecane", Martin Newcomb, John H. Horner, Michael T. Blanda, Chau Ha and Phillip J. Squatrito, November 28, 1990
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12. "Parameters for Force Field Calculations of Compounds Containing Tetrahedral Tin", Martin Newcomb and John H. Horner, December 10, 1990
13. "Macrocycles Containing Tin. Crystal Structures and Molecular Mechanics, Calculated Structures of Macrobicyclic Lewis Acidic Hosts", Martin Newcomb and John H. Horner, December 12, 1990

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1. "Macrocycles Containing Tin. Solid Complexes of Anions Encrypted in Macrobicyclic Lewis Acid Hosts", M. Newcomb, J. H. Horner, M. T. Blanda, P. J. Squattrito, *J. Am. Chem. Soc.* **111**, 6294-6301 (1989).
2. "Macrocycles Containing Tin. The Preparation of Macrobicyclic Lewis Acidic Hosts Containing Two Tin Atoms and  $^{119}\text{Sn}$  NMR Studies of Their Chloride and Bromide Binding Properties in Solution", M. T. Blanda, J. H. Horner, M. Newcomb, *J. Org. Chem.* **54**, 4626-4636 (1989).
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5. "Macrocycles Containing Tin. Crystal Structures and Molecular Mechanics Calculated Structures of Macrobicyclic Lewis Acidic Hosts", J. H. Horner, P. J. Squattrito, N. McGuire, J. P. Riebenspies, M. Newcomb, *Organometallics* **10**, 1741-1750 (1991).

**List of Personnel**

**Graduate Students**

Dr. Michael T. Blanda  
Mr. Paul W. Chambers  
Ms. Chau Ha  
Ms. Grace Ndip

**Post Doctoral Research Associates**

Dr. John H. Horner  
Dr. M. Udaya Kumar  
Dr. Nancy McQuire  
Dr. Phillip J. Squatritto

**Other Professional Personnel**

Dr. J. R. Riebenspies